

Form PTO-1390  
(Rev. 12-29-99)

US DEPARTMENT OF COMMERCE PATENT AND TRADEMARK OFFICE

ATTORNEY'S DOCKET NO

H 3497 PCT/US

TRANSMITTAL LETTER TO THE UNITED STATES  
DESIGNATED/ELECTED OFFICE (DO/EO/US)  
CONCERNING A FILING UNDER 35 U.S.C. 371

U.S. APPLICATION NO. (if known see 37 CFR 1.5)

09/856236

INTERNATIONAL APPLICATION NO.  
PCT/EP99/08645INTERNATIONAL FILING DATE  
November 10, 1999PRIORITY DATE CLAIMED  
November 17, 1998

## TITLE OF INVENTION

PREPARATION CONTAINING GYPSUM FROM FLUE GAS DESULPHURISATION, METHOD  
FOR USING SAME AND USE THEREOF

## APPLICANT(S) FOR DO/EO/US

Johann Klein, Gaby Schilling, Sabine Sipmann, Ralph Schuetze, Friedhelm Koepnick, Helmut Loth, Klaus  
Helpenstein, Wolfgang Klauck, Claudia Mai

Applicant herewith submits to the United States Designated/Elected Office (EO/DO/US) the following items and other information:

1. ☒ This is a **FIRST** submission of items concerning a filing under 35 U.S.C. 371
2. ☐ This is a **SECOND** or **SUBSEQUENT** submission of items concerning a filing under 35 U.S.C. 371.
3. ☐ This express request to begin national examination procedures (35 U.S.C. 371(f)) at any time rather than delay examination until the expiration of the applicable time limit set in 35 U.S.C. 371(b) and PCT Articles 22 and 39 (1).
4. ☒ A proper Demand for International Preliminary Examination was made by the 19th month from the earliest claimed priority date
5. ☒ A copy of the International Application as filed (35 U.S.C. 371(c)(2)).
  - a. ☐ is transmitted herewith (required only if not transmitted by the International Bureau)
  - b. ☒ has been transmitted by the International Bureau.
  - c. ☐ is not required, as the application was filed in the United States Receiving Office (RO/US).
6. ☒ A translation of the International Application into English (35 U.S.C. 371(c)(2)).
7. ☒ Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371(c)(3))
  - a. ☐ are transmitted herewith (required only if not transmitted by the International Bureau).
  - b. ☐ have been transmitted by the International Bureau.
  - c. ☐ have not been made; however, the time limit for making such amendments has NOT expired.
  - d. ☒ have not been made and will not be made.
8. ☐ A translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371(c)(3))
9. ☒ An oath or declaration of the inventor(s) (35 U.S.C. 371(c)(4)). UNEXECUTED
10. ☐ A translation of the annexes to the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371(c)(5)).

Items 11. to 16. below concern other document(s) or information included:

11. ☒ An Information Disclosure Statement under 37 CFR 1.97 and 1.98.
12. ☐ An assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included.
13. ☒ A **FIRST** preliminary amendment
  - ☐ A **SECOND** or **SUBSEQUENT** preliminary amendment.
14. ☐ A substitute specification.
15. ☐ A change of power of attorney and/or address letter.
16. ☒ Other items or information:

PTO Form 1449 (with references)  
Drawings (2 sheets)"Express Mail" mailing label number EL615775998US

U.S. Application No. (If known, see 37 CFR 1.53) <div style="font-size: 24pt; font-weight: bold; margin-top: -10px;">09/856236</div>	INTERNATIONAL APPLICATION NO. PCT/EP99/08645	ATTORNEY'S DOCKET NUMBER H 3497 PCT/US
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17. The following fees are submitted: <b>BASIC NATIONAL FEE (37 CFR 1.492(a)(1)-(5)):</b> Neither international preliminary examination fee (37 CFR 1.482) nor international search fee (37 CFR 1.445(a)(2)) paid to USPTO and International Search Report not prepared by the EPO or JPO..... <b>\$1000.00</b>  International preliminary examination fee (37 CFR 1.482) not paid to USPTO but International Search Report prepared by the EPO or JPO..... <b>\$860.00</b>  International preliminary examination fee (37 CFR 1.482) not paid to USPTO but international search fee (37 CFR 1.445(a)(2)) paid to USPTO ..... <b>\$710.00</b>  International preliminary examination fee paid to USPTO (37 CFR 1.482) but all claims did not satisfy provisions of PCT Article 33(1)-(4) ..... <b>\$690.00</b>  International preliminary examination fee paid to USPTO (37 CFR 1.482) and all claims satisfied provisions of PCT Article 33(1)-(4)..... <b>\$100.00</b>  <div style="text-align: right; font-weight: bold;">ENTER APPROPRIATE BASIC FEE AMOUNT                      =</div>	<div style="border-bottom: 1px solid black; margin-bottom: 5px;">CALCULATIONS</div> <div style="border-bottom: 1px solid black; margin-bottom: 5px;">PTO USE ONLY</div>
<div style="text-align: right; font-weight: bold;">ENTER APPROPRIATE BASIC FEE AMOUNT                      =</div>	<div style="border: 1px solid black; padding: 5px; width: 100%;"> <div style="display: flex; justify-content: space-between;"> <span>\$</span> <span>860</span> </div> </div>

Surcharge of <b>\$130.00</b> for furnishing the oath or declaration later than <input type="checkbox"/> 20 <input type="checkbox"/> 30 months from the earliest claimed priority date 37 (CFR 1.492(e)).				\$	0
CLAIMS	NUMBER FILED	NUMBER EXTRA	RATE		
Total Claims	1 - 20 =	0	0 X \$18.00	\$	0
Independent Claims	1 - 3 =	0	0 X \$80.00	\$	0
Multiple dependent claims (s)(if applicable)		0	+ \$270.00	\$	0
TOTAL OF ABOVE CALCULATIONS                      =				\$	860
Reduction of 1/2 for filing by small entity, if applicable. A Small Entity Statement must also be filed. (Note 37 CFR 1.9, 1.27, 1.28).				\$	0
SUBTOTAL                      =				\$	860
Processing fee of <b>\$130.00</b> for furnishing the English translation later the <input type="checkbox"/> 20 <input type="checkbox"/> 30 months from the earliest claimed priority date (37 CFR 1.492(f)).				\$	0
TOTAL NATIONAL FEE                      =				\$	860
Fee for recording the enclosed assignment (37 CFR 1.21(h)). The assignment must be accompanied by an appropriate cover sheet (37 CFR 3.28, 3.31). \$40.00 per property				\$	0
TOTAL FEES ENCLOSED                      =				\$	860

	Amount to be: refunded:	\$-----
	charged:	\$ 860.00

a. ☐ A check in the amount of \$\_\_\_\_\_ to cover the above fees is enclosed.

b. ☒ Please charge my Deposit Account No. 01-1250 in the amount of \$ 860.00 to cover the above fees.  
 A triplicate copy of this sheet is enclosed. Order No. 01-0392.

c. ☒ The Commissioner is hereby authorized to charge any additional fees which may be required, or credit any  
 overpayment to Deposit Account No. 01-1250. A duplicate copy of this sheet is enclosed.

**NOTE: Where an appropriate time limit under 37 CFR 1.494 or 1.495 has not been met, a petition to revive (37 CFR 1.137 (a) or (b))  
 must be filed and granted to restore the application to pending status.**

SEND ALL CORRESPONDENCE TO:      Henkel Corporation, Law Dept. 2500 Renaissance Blvd., Suite 200 Gulph Mills, PA 19406	<div style="text-align: center;">           SIGNATURE:       </div> <div style="margin-top: 20px;">         Kimberly R. Hild          NAME ATTORNEY FOR APPLICANT          39,224          REGISTRATION NUMBER       </div>
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PATENT

Docket No. H 3497 PCT/US

**IN THE UNITED STATES PATENT AND TRADEMARK OFFICE**

**In re Application of:**

Klein, et al.

**International Application No.**

PCT/EP99/08645

**International Filing Date:**

November 10, 1999

**Serial No.** 09/856,236

**Examiner:** To be assigned

**Filed:** To be assigned

**Art Unit:** To be assigned

**Title:** PREPARATION CONTAINING GYPSUM FROM FLUE GAS  
DESULPHURISATION, METHOD FOR USING SAME AND USE  
THEREOF

"Express Mail Post Office to Addressee" service mailing label number EL615774895US

**SUPPLEMENTAL PRELIMINARY AMENDMENT**

Box PCT  
Assistant Commissioner for Patents  
Washington, DC 20231

Attn: DO/EO/US

Sir:

Prior to examining this application, please amend the application as follows:

**In the Claims**

Please cancel Claim 1, without prejudice.

Please add the following new claims:

12. (New) A composition comprising:

(a) at least one water-soluble polymer or water-dispersible polymer, or combinations thereof; and

(b) one or more filler particles comprising WDP gypsum particles, wherein the WDP gypsum particles are obtained from waste-gas desulphurization plants and have a particle size distribution mean diameter ranging from 13  $\mu\text{m}$  to 500  $\mu\text{m}$ .

13. (New) The composition of claim 12, wherein the particle size distribution mean diameter of the WDP gypsum particles ranges from 30  $\mu\text{m}$  to 250  $\mu\text{m}$ .

14. (New) The composition of claim 13, wherein the filler particles comprise at least one other type of filler particles in addition to the WDP gypsum particles.

15. (New) The composition of claim 14, wherein the other type of filler particles comprise inorganic filler particles selected from the group consisting of chalk, titanium dioxide, barium sulfate, silica flour, silica gel, dolomite, kaolin and combinations thereof.

16. (New) The composition of claim 15, wherein the water-soluble or water dispersible polymer is selected from the group consisting of polyurethanes, polyacrylates, polymethacrylates, polyvinyl esters, polystyrenes, polybutadienes, polyamides, polyesters, polyvinyl chlorides, ethylene/vinyl acetate copolymers, styrene/butadiene copolymers, styrene/acrylonitrile polymers, styrene/acrylate copolymers and combinations thereof.

17. (New) The composition of claim 16, wherein the filler particles are present in the composition in a total amount of at least 40 weight percent, based on the total weight of the composition.

18. (New) The composition of claim 17, wherein the composition comprises from 50 weight percent to 99 weight percent of the WDP gypsum particles or a mixture of the WDP gypsum particles and the at least one other type of inorganic filler particles, from 1 weight percent to 50 weight percent of the water-soluble or water dispersible polymer, from 0 weight percent to 49 weight percent by weight of water, and from 0 weight percent to 49 weight percent of other additives.

19. (New) The composition of claim 12, wherein the filler particles, in addition to the WDP gypsum particles, comprise at least one other type of filler particles selected from the group consisting of chalk, titanium dioxide, barium sulfate, silica flour, silica gel, dolomite, kaolin and combinations thereof.

20. (New) The composition of claim 12, wherein the water-soluble or water dispersible polymer is selected from the group consisting of polyurethanes, polyacrylates, polymethacrylates, polyvinyl esters, polystyrenes, polybutadienes, polyamides, polyesters, polyvinyl chlorides, ethylene/vinyl acetate copolymers, styrene/butadiene copolymers, styrene/acrylonitrile polymers, styrene/acrylate copolymers and combinations thereof.

21. (New) The composition of claim 12 wherein the composition is a surface coating, a surfacing composition, a sealing composition, an adhesive, or a molding composition.

22. (New) A process for preparing a polymer-containing composition comprising combining in any order one or more water-soluble polymers or water-dispersible polymers, or combinations thereof with filler particles, wherein the filler particles comprise WDP gypsum particles that are obtained from waste-gas desulphurization plants and have a particle size distribution mean diameter ranging from 13  $\mu\text{m}$  to 500  $\mu\text{m}$  to form the polymer-containing composition.

23. (New) The process of claim 22 wherein the filler particles comprise a mixture of the WDP gypsum particles and at least one other type of inorganic filler particles.

24. (New) The process of claim 22 wherein the water-soluble polymers or water-dispersible polymers are in an aqueous dispersion prior to the combination with the filler particles.

25. (New) The process of claim 22 wherein the polymer-containing composition is in the form of a solid powder, a paste, an aqueous dispersion, or a non-aqueous liquid.

26. (New) The process of claim 22 wherein water or one or more other additives, or combinations thereof are combined in any order with the polymers and filler particles to form the polymer-containing composition.

27. (New) The process of claim 22 wherein the polymer-containing composition is a polymer dispersion and wherein the filler particles comprise at least one other type of inorganic filler particles and the WDP gypsum particles have a particle size distribution mean diameter ranging from 30  $\mu\text{m}$  to 250  $\mu\text{m}$ .

28. (New) A process for preparing a surface coating, a surfacing compound, a sealing compound, an adhesive, or a molding composition comprising combining one or more water-soluble or water-dispersible polymers with WDP gypsum particles wherein the WDP gypsum particles are obtained from waste-gas desulphurization plants and have a particle size distribution mean diameter ranging from 13  $\mu\text{m}$  to 500  $\mu\text{m}$  to form the surface coating, surfacing compound, sealing compound, adhesive, or molding composition.

29. (New) The process of claim 28 wherein the particle size distribution mean diameter of the WDP gypsum particles ranges from 30  $\mu\text{m}$  to 250  $\mu\text{m}$ .

30. (New) A surface coating, a surfacing compound, a sealing compound, an adhesive, or a molding composition prepared by the process of claim 28.

0905276-03404  
T07280-9829250

**REMARKS**

Applicants respectfully request the Examiner to enter the above amendments prior to examination of this application.

**Status of Claims**

Claims 12 to 30 will be pending after entry of the present amendment. Claim 1 is being canceled without prejudice.

**Amendment**

New Claims 12 to 30 replace original Claims 1 to 11, and are being presented to better conform with US patent practice. These new claims are supported by the specification for example as shown in the Table below (cites to the specification are for the English translation):

<b>Claim</b>	<b>Support in Specification</b>
12	page 3, line 25 to page 4, line 6
13	page 9, lines 23 to 24
14	page 10, lines 9 to 11
15, 19	page 10, line 28 to page 11, line 12
16, 20	page 5, lines 20 to 27
17	page 12, lines 11 to 15
18	page 12, lines 13 to 14, page 13, lines 6 to 13, original claim 7
21	page 14, lines 8 to 13
22, 23, 24, 26	page 13, lines 19 to 30
25	page 12, lines 16 to 25
27	page 14, lines 16 to 19
28	page 14, lines 8 to 13
29	page 14, lines 14 to 15
30	page 14, lines 8 to 13

No new matter is added by the new claims or amendments to the specification. The mean diameter of the WDP gypsum particles is measured with a Sympatec Helos H0720 in isopropanol.

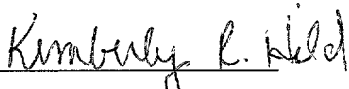


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CONCLUSION

Applicants respectfully request early and favorable notification of allowance of all pending claims. The Assistant Commissioner is authorized to charge any deficiency in the required fee or to credit any overpayment to Deposit Account 01-1250 in connection with this amendment.

Respectfully submitted,

  
\_\_\_\_\_  
Kimberly R. Hild  
(Reg. No. 39,224)  
Attorney for Applicants  
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Gulph Mills, PA 19406

PATENT

Docket No. H 3497 PCT/US

**IN THE UNITED STATES PATENT AND TRADEMARK OFFICE****In re Application of:**

Klein, et al.

**International Application No.**

PCT/EP99/08645

**International Filing Date:**

November 10, 1999

**Serial No.** To be assigned**Examiner:** To be assigned**Filed:** To be assigned**Art Unit:** To be assigned

**Title:** PREPARATION CONTAINING GYPSUM FROM FLUE GAS  
DESULPHURISATION, METHOD FOR USING SAME AND USE  
THEREOF

"Express Mail Post Office to Addressee" service mailing label number EL615775998US

**PRELIMINARY AMENDMENT**

Box PCT  
Assistant Commissioner for Patents  
Washington, DC 20231

Attn: DO/EO/US

Sir:

Prior to examining this application, please amend the application as follows:

**In the Specification (Using the English Translation):**

On page 1 of the English translation, on a separate line between the title and line 1, please insert the following header and paragraph on consecutive lines as shown below:

**-- CROSS REFERENCE TO RELATED APPLICATIONS**

This application is a national stage application under 35 U.S.C. § 371 of

**Docket No. H3497 PCT/US**  
**PCT/EP99/08645**

international application PCT/EP99/08645 filed on November 10, 1999, the international application not being published in English. This application also claims priority under 35 U.S.C. §119 to DE 198 53 006.4, filed on November 17, 1998. --

On page 1, on a separate line immediately after the above inserted paragraph and before line 1, please insert the following header:

-- FIELD OF THE INVENTION --.

On page 1, on a separate line between lines 5 and 6, please insert the following header:

-- BACKGROUND OF THE INVENTION -- .

On page 3, on a separate line between lines 24 and 25, please insert the following header:

-- SUMMARY OF THE INVENTION -- .

On page 3, on a separate line after line 30, please insert the following header and paragraph on consecutive lines:

-- BRIEF DESCRIPTION OF THE DRAWINGS

Fig. 1 is an electron micrograph showing WDP gypsum particles useful in the present invention.

Fig. 2 is a graph showing the particle size distribution of WDP gypsum particles useful in the present invention.

On page 4, on a separate line before line 1, please insert the following header:

-- DETAILED DESCRIPTION OF THE INVENTION --.

On page 19, line 1, please replace the heading "CLAIMS" with the following heading:

-- What is claimed is: --

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**PCT/EP99/08645**

On a separate page, after page 20, please insert the enclosed Abstract of the Disclosure.

**In the Claims**

Please cancel Claims 2 to 11, without prejudice.

**REMARKS**

Applicants respectfully request the Examiner to enter the above amendments prior to examination of this application.

**Amendment**

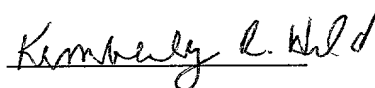
The specification is being amended to insert section headers and an abstract of the disclosure in accordance with 37 CFR §1.77 to better conform with US patent practice. The specification is also being amended to insert a cross-reference to related applications in accordance with 37 CFR §1.78 and to claim priority to those applications listed therein.

No new matter is added by the amendments to the specification.

**CONCLUSION**

The Assistant Commissioner is authorized to charge any deficiency in the required fee or to credit any overpayment to Deposit Account 01-1250 in connection with this amendment.

Respectfully submitted,



Kimberly R. Hild  
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**Abstract of the Disclosure**

The present invention relates to a composition, and a method of preparing and using the same. The composition contains at least one water-soluble or water-dispersible polymer or combinations thereof, and one or more filler particles that includes WDP gypsum particles. The WDP gypsum particles are obtained from waste gas-desulphurization plants and have a particles size distribution mean diameter ranging from 13  $\mu\text{m}$  to 500  $\mu\text{m}$ . The composition is particularly useful as a surface coating, a surfacing compound, a sealing compound, an adhesive or a molding composition.

Preparation Containing Gypsum from Flue Gas Desulphurization, Method  
for Using Same and Use Thereof

This invention relates to a preparation containing a water-soluble polymer or a water-dispersible polymer or a mixture of two or more thereof and gypsum particles from waste-gas desulfurization plants (WDP gypsum) with a value for the particle size distribution x50 of 13 to 500  $\mu\text{m}$ , to a  
5 process for its production and to its use.

Inorganic inert fillers and polymeric materials are often used in admixture with one another. Depending on the ratio by weight of filler to polymeric material, properties can be attributed to the end product obtainable from such a mixture that would be very difficult or even  
10 impossible to achieve using a material which consisted of only one of the components of the mixture (i.e. only polymer or only filler). The combination of inorganic inert filler and polymers is often aimed at and used not least because both materials have very different property profiles of which the combination is not only desirable but actually necessary for  
15 many applications.

Fillers which generally consist of a number of individual loose filler particles are often chemically inert to the environment around them. Because of this, filler particles can often only be formed/shaped together with a binder. Suitable binders are, for example, organic and inorganic  
20 binders. One exception to this are fillers which are capable of setting to form solid materials by reacting with a reactant present in the surrounding environment. Examples of such fillers are gypsum in the form of anhydrite and various lime compounds which are capable of curing by reacting with water or carbon dioxide from the surrounding air.

25 Whereas the self-setting fillers mentioned above generally cure to form brittle hard materials, a distinctly broader spectrum of physical and



chemical properties can often be achieved by replacing the inorganic materials with polymers. However, disadvantages of using polymers only generally include increased production costs by comparison with inorganic materials and their lower hardness and chemical resistance, particularly in regard to fire resistance and flameproofing.

In this connection, the building supplies industry in particular has a steadily increasing demand for new materials which combine the positive properties of fillers, such as their chemical resistance, their resistance to heat, their availability in large quantities and their low price, with those of the polymers. The demand ranges from surface coating compositions or adhesives, which are generally applied as a thin layer to surfaces of various kinds, through surfacing and sealing compounds to plastics of the type used, for example, as a sheathing for power cables or as water conduits.

For example, Wirsching, Hüller, Hofmann and Pürzer describe the use of fillers of WDP gypsum in ZKG INTERNATIONAL, No. 5, 1995 (Vol. 48), pp. 241-256 (Bauverlag GmbH). The article in question describes in particular the use of WDP gypsum from coal-fired power stations in adhesives, paints and plastics. Before being used as a filler, the gypsum is finely ground so that it has a mean particle diameter of about 8 to 12  $\mu\text{m}$  and the upper cut of the particle diameter is at about 25 to 50  $\mu\text{m}$ .

JP 76-139114 relates to the use of WDP gypsum as a pigment in coating compositions. This document describes a composition of titanium dioxide, WDP gypsum, aluminium silicate, ethylene/vinyl acetate copolymer, polyvinyl acetate, thickener and water as a white emulsion which is suitable as a coating composition.

Filler-containing polymer materials are often marketed and processed in the form of aqueous dispersions. Unfortunately, such materials frequently have serious disadvantages during and after processing. Firstly, the viscosity of the dispersions often cannot be



adjusted to a value suitable for processing. Secondly, after processing and generally after drying, the filler-containing polymer material applied undergoes a distinct change in volume in relation to its volume at the time of application. This change in volume can often correspond to the extent to  
5 be expected as a result of evaporation of the water present in the dispersion.

Behavior such as this (often referred to as "shrinkage" or "contraction") is undesirable, above all for filler-containing polymer dispersions which are supposed to perform a "filling" function. Thus, in the  
10 case of surface coatings for example, importance is often attributed to the levelling of any structural unevenness of the substrate. Surfacing or sealing compounds, for example, are expected to fill the void to be sealed or filled - even after drying of the compound introduced - to the full extent to which the dispersion was originally introduced.

15 In addition, the contraction of surfacing compounds frequently leads during drying of the compound to the formation of cracks therein which, besides the generally noticeable visual disadvantages, often represent starting points for the penetration of corrosive compounds or moisture. The visual impression and the stability of the region thus filled are often  
20 drastically impaired as a result.

Filler-containing polymer materials are often used as adhesives, more particularly emulsion-based adhesives. Unfortunately, many adhesives of this type lack elasticity which is often a disadvantage to the stability of the adhesive bond under stress.

25 Now, the problem addressed by the invention was to remedy these disadvantages. This problem has been solved by a polymer composition which, besides a water-soluble or water-dispersible polymer or a mixture of two or more such polymers, contains filler particles in the form of gypsum particles from waste-gas desulfurization plants of which the particle size  
30 distribution has a mean diameter ( $x_{50}$ ) of about 13 to about 500  $\mu\text{m}$ .

Accordingly, the present invention relates to a preparation containing a water-soluble polymer or a water-dispersible polymer or a mixture of two or more thereof and filler particles, the filler particles being gypsum particles from waste-gas desulfurization plants (WDP gypsum) with a value  
5 for the particle size distribution x50 of 13 to 500  $\mu\text{m}$  (as measured with a Sympatec Helos H0720 in isopropanol).

In the context of the invention, a "preparation" is any mixture which contains the constituents mentioned above. It may be a mixture which is already in a form suited to the application envisaged (for example already  
10 provided with a suitable quantity of water) or which first has to be brought by the user into a form suitable for application, for example a powder dispersible in water.

A "water-soluble polymer or water-dispersible polymer" in the context of the present invention is a polymer which, in the form of a mixture  
15 with water, forms a molecularly disperse solution, a substantially stable gel or colloid or a substantially stable dispersion. In the context of the present invention, it does not matter whether the stability of the aqueous preparations mentioned derives from the properties of the polymer itself or is supported by such additives as emulsifiers, stabilizers, gel formers or the  
20 like.

The preparation according to the invention may contain, for example, only a certain water-soluble polymer or a certain water-dispersible polymer. However, the preparation may equally well contain a mixture of two or more water-soluble polymers or a mixture of two or more  
25 water-dispersible polymers. According to the invention, the preparation may also be a mixture of one or more water-soluble polymers and one or more water-dispersible polymers.

The water solubility or self-dispersibility of polymers can be based, for example, on the presence of anionic or cationic groups such as are  
30 normally present in polymers to achieve such an effect. Solubility or

dispersibility in water can also be obtained by arranging nonionic groups on a polymer suitable for use in accordance with the invention, these nonionic groups leading to water solubility or water dispersibility of the polymer.

Suitable cationic groups are, for example, quaternized amino groups; suitable anionic groups are, in particular, acid groups.

For example, water-soluble polymers obtainable by polymerization of monomer constituents which impart water solubility to the polymers may be used for the purposes of the present invention. Such polymers include, for example, the polymers of acrylic acid and the polymers obtainable by polyaddition of alkylene oxides. Polymers which are "self-dispersible" in water are also suitable. "Self-dispersible polymers" are understood to be polymers which form a substantially stable dispersion in water without the addition of emulsifiers. In general, polymers such as these contain, for example, carboxylic acid groups, sulfonic acid groups, phosphonic acid groups or chain segments of polyethylene oxide or a mixture of two or more of the functional groups mentioned as functional groups.

Polymers which are neither soluble in water nor "self-dispersible" may be converted into a substantially stable emulsion or dispersion in water, for example using commercially available emulsifiers or dispersants.

Suitable polymers are, for example, polyurethanes, polyacrylates, polymethacrylates, polyvinyl esters, polystyrene and sulfonated polystyrene, polybutadiene and sulfonated polybutadiene, polyamides, polyesters and polyvinyl chloride. Other suitable polymers are corresponding co- and ter-polymers, such as ethylene/vinyl acetate copolymers (EVA), styrene/butadiene copolymers (SBR), styrene/acrylonitrile polymers (SAN), styrene/acrylate copolymers and the like. Another preferred embodiment is characterized by the use of, for example, polymers obtainable from the polymerization of acrylates or from the co- and ter-polymerization of acrylates with acrylonitrile, vinyl esters, maleates, acrylic acid, styrene and the like. Polymers such as these and

polymers dispersions resulting from them are comprehensively described, for example, in "Encyclopaedia of Polymer Science and Technology" (Editors: Mark, Bikales, Overberger, Menges, 2nd Edition, 1989, Wiley, New York, 17, pp. 406-409).

5 In another preferred embodiment, water-soluble or water-dispersible polymers or mixtures thereof already present in dissolved or dispersed form are used for the production of the preparation according to the invention. These polymers are in particular aqueous dispersions of synthetic polymers of the type mentioned above, more particularly polyurethanes, 10 poly(meth)acrylates, polyvinyl esters, polystyrene, polybutadiene, polyamides or polyvinyl chloride, or mixtures of two or more thereof. The corresponding co- and ter-polymers, styrene/butadiene, styrene/acrylates may also be used for the purposes of the present invention, as may natural latices. The dispersions usable in accordance with the invention may be 15 prepared, for example, by suspension or emulsion polymerization of the corresponding monomers. Secondary dispersions - obtainable by dispersing a polymer melt in a suitable medium - may also be used.

Polymer dispersions which are generally obtainable commercially in large quantities as the outcome of suspension or emulsion polymerizations 20 and which are used, for example, as binders for emulsion paints or emulsion-based adhesives (see, for example, Römpp Chemie-Lexikon, Vol. 2, Thieme-Verlag, 1990, pp. 1010-1011 and the literature cited therein) are preferably used. Unsaturated, radical-polymerizable compounds, such as acrylates and methacrylates, dienes or olefins, or mixtures of two or 25 more thereof are particularly suitable as monomers for such polymer dispersions. The suspension or emulsion polymerization is described, for example, in "Ullmann's Enzyklopädie der technischen Chemie" (Vol. A21, 5th Edition, VCH, 1987), reference being expressly made to this literature reference.

30 Polymer dispersions used in a preferred embodiment of the present

invention are based on polyvinyl esters, such as polyvinyl acetate, and co- and ter-polymers of the vinyl esters with monomers, such as ethylene, acrylates or methacrylates or maleic acid mono- and diesters, or mixtures of two or more thereof. Monomers which lead to dispersions suitable for use in accordance with the present invention are described, for example, in Ullmann's Enzyklopädie der technischen Chemie (Vol. A22, 1993, VCH, pp. 1-15). Dispersions prepared on the basis of such monomers are described, for example, in "Handbook of Additives" (3rd Edition, Chapman and Hall, pp. 381-399). Reference is hereby expressly made to the cited literature references.

Polymers suitable in a preferred embodiment are, for example, the copolymers of vinyl acetate, vinyl propionate or VeoVa® 9 or 10 with other comonomers. VeoVa® 9 and 10 are the vinyl esters of tert. carboxylic acids (Versatic® Acid 9 or 10) for copolymerization with, for example, vinyl acetate for emulsion paints, plasters, concrete additives, paper and textile coatings, emulsion-based adhesives and paints (manufacturer: Deutsche Shell Chemie). Particularly suitable polymers are co- and ter-polymers containing vinyl acetate/dibutyl maleate, vinyl acetate/n-butyl acrylate, vinyl acetate/2-ethylhexyl acrylate, vinyl acetate/n-butyl acrylate/N-hydroxymethyl acrylamide, vinyl acetate/crotonic acid, vinyl acetate/VeoVa® 10, vinyl acetate/VeoVa® 10/acrylic acid, vinyl acetate/VeoVa® 10/n-butyl acrylate, vinyl acetate/N-hydroxymethyl acrylamide, vinyl acetate/vinyl laurate, vinyl acetate/vinyl laurate/vinyl chloride, vinyl acetate/ethylene/vinyl chloride, vinyl acetate/ethylene/acrylate, vinyl acetate/ethylene/acrylamide, vinyl acetate/ethylene/N-hydroxymethyl acrylamide, vinyl propionate, vinyl propionate/vinyl chloride, vinyl propionate/tert.butyl acrylate, VeoVa® 10/vinyl chloride, VeoVa® 10/styrene/acrylate, VeoVa® 10/styrene/maleate, VeoVa® 10/styrene/acrylate/maleate and VeoVa® 10/VeoVa® 9/methyl methacrylate/butyl acrylate, VeoVa® 10 being completely or partly

replaceable by VeoVa® 9.

Another preferred embodiment of the invention is characterized by the use of polymer dispersions based on poly(meth)acrylates and co- and ter-polymers of the (meth)acrylates with such monomers as acrylonitrile, vinyl esters, maleates, acrylic acid and styrene. Corresponding polymer dispersions are comprehensively described, for example, in "Emulsion Polymerisation and Emulsion Polymers" (1997), John Wiley, pp. 619-655, New York) and are regarded as part of the present disclosure.

Another preferred embodiment of the invention is characterized by the use of commercially obtainable dispersions, such as DL 345 (manufacturer: Dow Latex) or Acronal® DS 3518 (manufacturer: BASF AG).

Aqueous polymer dispersions based on conjugated dienes, such as chloroprene or butadiene, and copolymers of these dienes with unsaturated compounds, such as styrene or acrylonitrile, may also be used in accordance with the present invention. Such dispersions are known and are described, for example, in "Emulsion Polymerisation and Emulsion Polymers" (1997), John Wiley, pp. 521-561, New York). Reference is hereby specifically made to the cited literature reference.

Besides the polymers mentioned, monomers with additional functional groups, such as N-methylol acrylamide, hydroxypropyl acrylate, (meth)acrylic acid or a mixture of two or more of the compounds mentioned, may be used for preparing the polymer dispersions usable in accordance with the invention.

The preparations according to the invention contain at least WDP gypsum particles with a value for the particle size distribution x50 of 13 to 500 µm (as measured with a Sympatec Helos H0720) as filler.

WDP gypsum particles differing in their dimensions are formed in different waste-gas desulfurization plants in dependence upon the technical parameters. It has been found in accordance with the present invention

that the advantages according to the invention can be obtained with WDP gypsum particles having the above-mentioned values for the particle size distribution.

In general, various processes may be used to measure a particle size distribution and the corresponding value  $x_{50}$ . Typical processes include, for example, the sieve process where a certain quantity of particles is sieved through sieves with different mesh widths. The total quantity of particles is thus divided into fractions with different particle diameters of which the quantity is expressed as a percentage of the total weight of the particles investigated. Other methods for determining particle size distribution are, for example, light scattering and Fraunhofer diffraction. In the context of the present invention, the particle size distribution of the WDP gypsum particles was determined by the Fraunhofer diffraction technique using a Sympatec Helos H0720. The particle size distribution was measured on a suspension in isopropanol. The following particle size distribution data are based on measurements by the technique mentioned but are not confined to such measurements. The advantages according to the invention can generally be obtained with any WDP gypsum particles whose particle size distribution is largely within the range mentioned, irrespective of the measuring system.

In a preferred embodiment of the present invention, the WDP gypsum particles have a lower limit to the particle size  $x_{50}$  of at least about 25  $\mu\text{m}$ . In another preferred embodiment, the value for the particle size distribution  $x_{50}$  is about 30 to 250  $\mu\text{m}$ . Good results can be obtained, for example, where the values for the particle size distribution  $x_{50}$  are in the range from about 35 to about 200 or of the order of 150  $\mu\text{m}$ . Another preferred embodiment of the invention is characterized by the use of WDP gypsum particles with a  $x_{50}$  value for the particle size distribution of from about 40 to about 120  $\mu\text{m}$ , for example in the range from about 60 to about 110  $\mu\text{m}$  and more particularly in the range from about 80 to about 100  $\mu\text{m}$ .

It has also been found to be of advantage for the filler particles to have a granular or rodlet-like form.

The WDP gypsum particles used as filler particles in accordance with the invention show their advantages according to the invention even when they are used as sole filler. In this case, WDP gypsum particles with a x50 value for the particle size distribution in the range from about 13 to about 110  $\mu\text{m}$  and more especially in the range from about 35 to about 80  $\mu\text{m}$  are used in a preferred embodiment of the invention.

In another preferred embodiment of the present invention, the WDP gypsum particles are used in admixture with at least one other type of inorganic filler particles.

In the context of the invention, the expression "one other type" of inorganic filler particles is intended to encompass any filler particles which differ from WDP gypsum particles either in their chemical composition, in their predominant spatial form (for example their crystal form) or in a combination of two or more of the features mentioned. In a preferred embodiment of the present invention, filler particles which differ at least in the x50 value of their particle size distribution from the corresponding value of the particle size distribution of the WDP gypsum particles are used as the other type of inorganic filler particles.

Suitable other types of inorganic filler particles are, for example, any inorganic filler particles inert to the other substances present in the preparation according to the invention. So far as the other type of inorganic filler particles is concerned, there are no restrictions on the x50 value for the particle size distribution. For example, filler particles with a x50 value for the particle size distribution in the range from about 0.01 to about 500  $\mu\text{m}$  may be used in accordance with the present invention.

Suitable other types of inorganic filler particles are, for example, filler particles of andalusite, sillimanite, kyanite, pyrophyllite, imigolite or allophane. Also suitable are compounds based on sodium aluminates or



calcium silicates and minerals, such as silica, calcium sulfate (gypsum) which does not come from waste-gas desulfurization plants in the form of anhydrite, semihydrate or dihydrate, silica flour, silica gel, barium sulfate, titanium dioxide, zeolites, leucite, potash feldspar, biotite, the group of  
5 soro-, cyclo-, ino-, phyllo- and tectosilicates, the group of poorly soluble sulfates, such as gypsum, anhydrite or heavy spar, and calcium minerals, such as calcite or chalk ( $\text{CaCO}_3$ ). According to the invention, the inorganic materials mentioned may be used individually as another type of inorganic  
10 filler particles. However, a mixture of two or more of the compounds mentioned may equally well be used. A preferred embodiment of the invention is characterized by the use of calcite, kaolin, dolomite, silica flour and gypsum ( $\text{CaSO}_4 \cdot 2 \text{H}_2\text{O}$ ).

In another preferred embodiment of the present invention, the filler particles of the other type have a  $x_{50}$  value for the mean particle size  
15 distribution in the range from about 1 to about 120  $\mu\text{m}$ , for example in the range from about 3 to about 60  $\mu\text{m}$  or in the range from about 60 to about 90  $\mu\text{m}$ .

Organic filler particles which cannot readily be assigned to the water-soluble or water-dispersible polymers are also suitable for use as  
20 another type of filler particles. These include in particular the finely ground plastic powders obtained in the recycling of plastics, more particularly the plastic powders obtainable from the fine grinding of highly crosslinked elastomeric or thermoset polymers. One example of such powders is the rubber powder obtained, for example, in the fine grinding of car tires.

Where the preparation according to the invention contains some  
25 filler particles of the other type either individually or in the form of a mixture of two or more thereof, the ratio by weight of WDP gypsum particles to filler particles of the other type is about 1:1,000 to about 1,000:1. Good results can be obtained, for example, if the ratio by weight of WDP gypsum  
30 particles to filler particles of the other type is about 1:10 to about 10:1 and

more particularly about 5:1 to about 1:5.

In another preferred embodiment of the present invention, the filler component of the preparation contains at least about 0.5% by weight and, more particularly, at least about 1% by weight of WDP gypsum particles. In

5 another preferred embodiment of the present invention, the percentage content of WDP gypsum particles is at least about 10% by weight or at least about 20% by weight. Good results are also obtained with percentage contents of WDP gypsum particles of at least about 30, 40 or 50% by weight or more, for example about 60 to 80% by weight or at least  
10 about 90% by weight.

In a preferred embodiment, the preparation according to the invention contains at least about 20% by weight of filler particles (WDP gypsum particles or other filler particles or a mixture thereof). A larger percentage of filler particles, for example about 30, 40 or 50 to 99% by  
15 weight, for example about 60 to 90% by weight, may also be added.

The compositions according to the invention may be present as a ready-to-use aqueous dispersion, i.e. they may contain the water-soluble or water-dispersible polymer or the mixture of two or more such polymers and the fillers together with water. According to the invention, however, the  
20 compositions may equally well contain no water or only a little water, i.e. for example may be present as a dry powder or as a paste of low water content. According to the invention, the preparation according to the invention may also be present as a water-free paste, a non-aqueous liquid, for example a solvent or another constituent of the preparation according to  
25 the invention, being used to establish the paste-like properties. Corresponding powders or pastes are of advantage, for example, when the actual preparation of an aqueous dispersion intended for use is to be left to the user himself/herself. In a preferred embodiment of the present invention, the preparation according to the invention is present in the form  
30 of a powder (redispersion powder).

If WDP gypsum particles are to be used as filler in accordance with the present invention, it is important to test the stability of the polymer dispersion used to calcium ions. In such a case, stability may have to be restored or improved by adding another emulsifier or dispersant or a mixture of two or more emulsifiers or dispersants.

Besides the polymer(s) and the filler particles, the preparation according to the invention may contain other ingredients. If the preparation according to the invention is intended to be ready-to-use, it may contain water. Depending on the application envisaged for the preparation according to the invention, the water content may vary between about 0 and 49% by weight.

Basically, water contents (based on the preparation as a whole) of about 0 to 49% by weight are suitable.

In addition to the constituents mentioned, the composition according to the invention may also contain one or more other additives.

Suitable other additives are, for example, emulsifiers, dispersants, stabilizers, defoamers, antioxidants, photostabilizers, pigment dispersants and the like.

The present invention also relates to a process for the production of a preparation containing a water-soluble polymer or a water-dispersible polymer or a mixture thereof and WDP gypsum particles with a particle size x50 of 13 to 500  $\mu\text{m}$  (as measured with a Sympatec Helos H0720 in isopropanol) or a mixture of WDP gypsum particles and at least one other type of inorganic filler particles, characterized in that at least one water-soluble polymer or at least one water-dispersible polymer or a mixture of two or more thereof or an aqueous dispersion containing one or more such polymers is mixed with WDP gypsum particles having a particle size x50 of 13 to 500  $\mu\text{m}$  (as measured with a Sympatec Helos H0720 in isopropanol) or a mixture of WDP gypsum particles and at least one other type of inorganic filler particles and optionally with water and one or more other

additives in one or more mixing steps in any order and with any time intervals between individual mixing steps.

The invention is illustrated by the accompanying drawings wherein:

Figure 1 is an electron micrograph of WDP gypsum with a granular  
5 to rodlet-like particle form.

Figure 2 shows the size distribution of the filler particles of a WDP gypsum used by way of example as filler.

The present invention also relates to the use of WDP gypsum particles having a x50 value of the particle size distribution of 13 to 500  $\mu\text{m}$   
10 (as measured with a Sympatec Helos H0720 in isopropanol) for the production of surface coatings, surfacing compounds, sealing compounds, adhesives or moldings with a content of water-soluble or water-dispersible polymers.

In a preferred embodiment of the invention, the WDP gypsum  
15 particles have a x50 value for the particle size distribution of 30 to 250  $\mu\text{m}$ .

The present invention also relates to the use of a mixture of inorganic filler particles containing WDP gypsum particles with a x50 value for the particle size distribution of 30 to 250  $\mu\text{m}$  and at least one other type of inorganic filler particles as a filler in polymer dispersions.

20 The invention is illustrated by the following Examples.

### Examples

#### Example 1: elasticity of emulsion-based adhesives

25 Compositions according to the invention show favorable elongation behavior when used as an emulsion-based adhesive.

Formulation:

acrylate dispersion (for example Acronal® DS 3518, BASF)	55 g
pigment dispersant (for example Pigmentverteiler A, BASF)	2 g

30 Fillers:

- A: fine calcite filler (for example Omyacarb 5GU, D50 value 6  $\mu\text{m}$ ; Omya)
- B: unground WDP gypsum (for example from Rethmann, D50 value 40  $\mu\text{m}$ )
- C: ground natural gypsum (for example Alabaster Brilliantweiß, D50 value 11  $\mu\text{m}$ ; Börgardts)
- D: coarse calcite filler (for example 50:50 mixture of Omyacarb 130 AL/Omyacarb 40 GU, D50 value (mixture) 88  $\mu\text{m}$ ; Omya)

Same-weight Filler Exchange							
Filler mixture					Max. force [kN/mm <sup>2</sup> ]	Elongation at max. force [%]	Elongation at break [%]
A [%]	A [g]	B [g]	C [g]	D [g]			
0	0	84	-	-	0.01	260	2840
50	42	42	-	-	0.02	680	1060
100	84	0	-	-	0.03	190	830
0.02	0	-	84	-	0.02	90	1050
0.03	42	-	42	-	0.03	140	900
100	84	-	0	-	0.03	190	830
0	0	-	-	125	0.01	420	2330
20	25	-	-	100	0.02	390	1500
50	62.5	-	-	62.5	0.02	340	1250
80	100	-	-	25	0.03	280	780
100	125	-	-	0	0.03	130	630

Example 2: volume shrinkage of surfacing compounds as a function of the filler composition

Visual evaluation

Formulation:

Styrene/acrylate dispersion (for example DL 345 from Dow Latex) 110 g

Pigment dispersant (for example Pigmentverteiler A, BASF) 4 g

Filler:

Filler used in 50 or 100% same-volume or same-weight exchange for filler

A (visual evaluation: -)

- A: fine calcite filler (for example Omyacarb 5GU, D50 value 6  $\mu\text{m}$ ; Omya)
- B: unground WDP gypsum (for example from Rethmann, D50 value 40  $\mu\text{m}$ )
- C: ground natural gypsum (for example Alabaster Brilliantweiß, D50 value 11  $\mu\text{m}$ ; Börgardts)
- D: coarse calcite filler (for example 50:50 mixture of Omyacarb 130 AL/Omyacarb 40 GU, D50 value (mixture) 88  $\mu\text{m}$ ; Omya)
- E: unground WDP gypsum (for example No. 1 from ProMineral, D50 value 36  $\mu\text{m}$ )
- F: unground WDP gypsum (for example No. 2 from ProMineral, D50 value 96  $\mu\text{m}$ )
- G: medium-coarse calcite filler (for example Omyacarb 40 GU, D50 value 44  $\mu\text{m}$ ; Omya)

- +: no visible volume shrinkage, no cracks
- $\pm$ : slight volume shrinkage/crack formation
- : distinct volume shrinkage/crack formation

Filler =	Same-weight Filler Exchange		Same-volume Filler Exchange	
	50% by weight X	100% by weight X	50% by vol. X	100% by vol. X
B	$\pm$	+	-	$\pm$
C	$\pm$	$\pm$	-	-
D	-	-	-	-
E	-	+	-	+
F	+	+	+	+
G	-	-	-	-

Example 3: tensile shear strength (wood-to-wood bonds) and viscosity behavior of emulsion-based adhesives

Formulation:

Styrene/acrylate dispersion (for example DL 345 from Dow Latex) 110 g

Pigment dispersant (for example Pigmentverteiler A, BASF) 4 g

Fillers:

A: fine calcite filler (for example Omyacarb 5GU, D50 value 6  $\mu\text{m}$ ; Omya)

B: unground WDP gypsum (for example from Rethmann, D50 value 40  $\mu\text{m}$ )

C: ground natural gypsum (for example Alabaster Brilliantweiß, D50 value 11  $\mu\text{m}$ ; Börgardts)

D: coarse calcite filler (for example 50:50 mixture of Omyacarb 130 AL/Omyacarb 40 GU, D50 value (mixture) 88  $\mu\text{m}$ ; Omya)

Same-weight Filler Exchange						
Filler Mixture					Tensile shear strength [N/mm <sup>2</sup> ]	Viscosity [scale units]
A [%]	A [g]	B [g]	C [g]	D [g]		
0	0	250	-	-	2.1	100
50	125	125	-	-	4.7	38
100	250	0	-	-	2.7	210
0	0	-	250	-	3.7	100
50	125	-	125	-	3.0	94
100	250	-	0	-	2.7	210
0	0	-	-	250	3.1	36
20	50	-	-	200	3.2	41
50	125	-	-	125	3.8	39
80	200	-	-	50	2.9	135
100	250	-	-	0	2.7	210

Example 4: Tensile shear strength (wood-to-wood bonds) and viscosity behavior of emulsion-based adhesives containing WDP gypsum with different particle size distributions

## Formulation:

Styrene/acrylate dispersion (for example DL 345 from Dow Latex) 110 g

Pigment dispersant (for example Pigmentverteiler A, BASF) 4 g

## Fillers:

A: fine calcite filler (for example Omyacarb 5GU, D50 value 6 µm; Omya)

E: unground WDP gypsum (for example No. 1 from ProMineral, D50 value 36 µm)

F: unground WDP gypsum (for example No. 2 from ProMineral, D50 value 96 µm)

G: medium-coarse calcite filler (for example Omyacarb 40 GU, D50 value 44 µm; Omya)

Same-weight Filler Exchange						
Filler Mixture					Tensile shear strength [N/mm <sup>2</sup> ]	Viscosity [scale units]
A [%]	A [g]	E [g]	F [g]	G [g]		
0	0	250	-	-	2.2	90
20	50	200	-	-	2.9	37
50	125	125	-	-	3.5	27
80	200	50	-	-	3.4	43
100	250	0	-	-	2.7	210
205	0	-	250	-	1.1	120
164	50	-	200	-	1.6	60
102	125	-	125	-	2.7	19
41	200	-	50	-	4.3	42
0	250	-	0	-	2.7	210
0	0	-	-	250	3.4	38
20	50	-	-	200	3.5	45
50	125	-	-	125	3.1	56
80	200	-	-	50	2.5	80
100	250	-	-	0	2.7	210



## CLAIMS

1. A preparation containing a water-soluble polymer or a water-dispersible polymer or a mixture of two or more thereof and filler particles, the filler particles being gypsum particles from waste-gas desulfurization plants (WDP gypsum particles) with a value for the particle size distribution x50 of 13 to 500  $\mu\text{m}$  (as measured with a Sympatec Helos H0720 in isopropanol).  
5
2. A preparation as claimed in claim 1, characterized in that the value for the particle size distribution x50 of the WDP gypsum particles is 30  $\mu\text{m}$  to 250  $\mu\text{m}$ .  
10
3. A preparation as claimed in claim 1 or 2, characterized in that it contains WDP gypsum particles in admixture with at least one other type of filler particles.
4. A preparation as claimed in claim 4, characterized in that inorganic  
15 filler particles selected from the group consisting of chalk ( $\text{CaCO}_3$ ), titanium dioxide, barium sulfate, silica flour, silica gel, dolomite or kaolin or a mixture of two or more thereof are present as the other type of filler particles.
5. A preparation as claimed in any of claims 1 to 4, characterized in that a polymer selected from the group consisting of polyurethanes,  
20 polyacrylates, polymethacrylates, polyvinyl esters, polystyrenes, polybutadienes, polyamides, polyesters, polyvinyl chlorides, ethylene/vinyl acetate copolymers (EVA), styrene/butadiene copolymers (SBR), styrene/acrylonitrile polymers (SAN), styrene/acrylate copolymers or a mixture of two or more thereof is present as the water-soluble or water-  
25 dispersible polymer.
6. A preparation as claimed in any of claims 1 to 5, characterized in that it contains at least 40% by weight of filler particles.
7. A preparation as claimed in any of claims 1 to 6, characterized in that it contains 50 to 99% by weight of WDP gypsum particles or a mixture  
30 containing WDP gypsum particles and at least one other type of inorganic

filler particles, 1 to 50% by weight of polymer, 0 to 49% by weight of water and 0 to 49% by weight of other additives

8. A process for the production of a preparation containing a water-soluble polymer or a water-dispersible polymer or a mixture thereof and
- 5 WDP gypsum particles with a particle size x50 of 13 to 500  $\mu\text{m}$  (as measured with a Sympatec Helos H0720 in isopropanol) or a mixture of WDP gypsum particles and at least one other type of inorganic filler particles, characterized in that at least one water-soluble polymer or at least one water-dispersible polymer or a mixture of two or more thereof or
- 10 an aqueous dispersion containing one or more such polymers is mixed with WDP gypsum particles having a particle size x50 of 13 to 500  $\mu\text{m}$  (as measured with a Sympatec Helos H0720 in isopropanol) or a mixture of WDP gypsum particles and at least one other type of inorganic filler particles and optionally with water and one or more other additives in one
- 15 or more mixing steps in any order and with any time intervals between individual mixing steps.
9. The use of WDP gypsum particles having a x50 value of the particle size distribution of 13 to 500  $\mu\text{m}$  (as measured with a Sympatec Helos H0720 in isopropanol) for the production of surface coatings, surfacing
- 20 compounds, sealing compounds, adhesives or moldings with a content of water-soluble or water-dispersible polymers.
10. The use claimed in claim 9, characterized in that the WDP gypsum particles have a x50 value for the particle size distribution of 30 to 250  $\mu\text{m}$ .
11. The use of a mixture of inorganic filler particles containing WDP
- 25 gypsum particles with a x50 value for the particle size distribution of 30 to 250  $\mu\text{m}$  and at least one other type of inorganic filler particles as a filler in polymer dispersions.

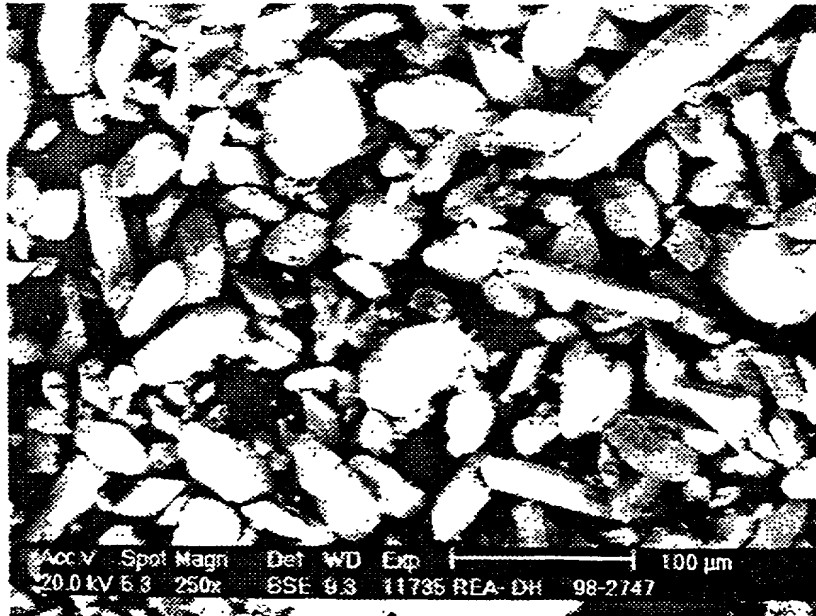
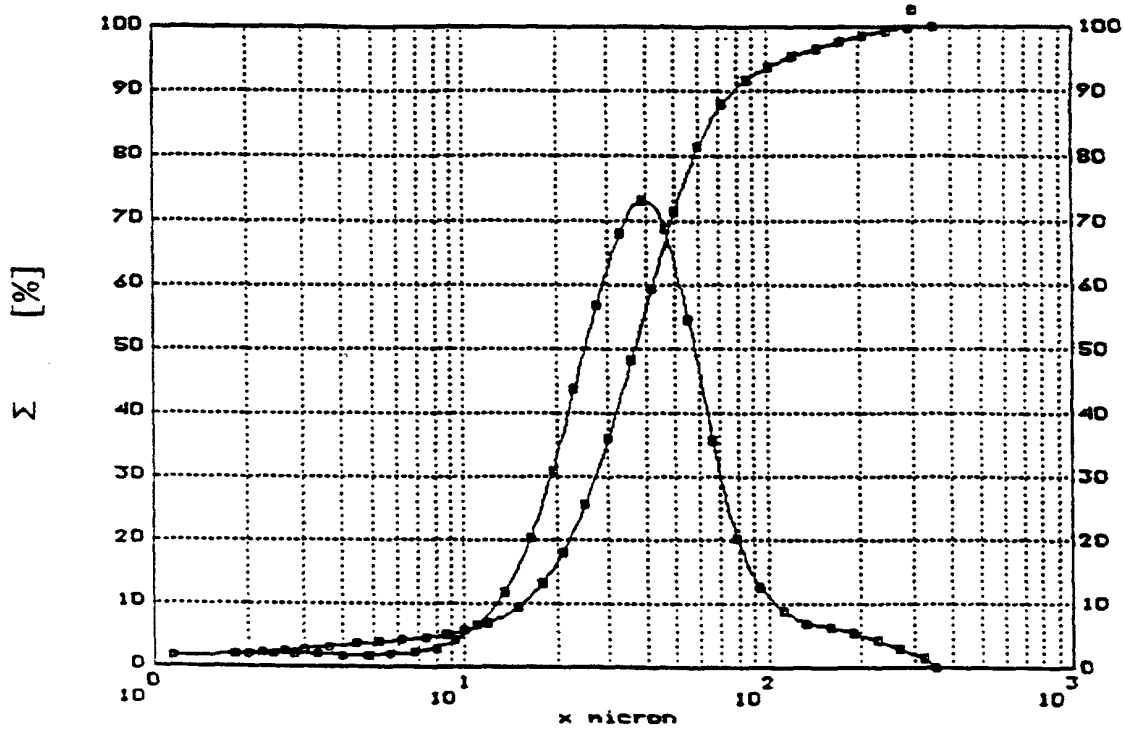


Fig. 1

Fig. 2



"Express Mail" mailing label number **EL615774895US**

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<b>DECLARATION FOR UTILITY OR DESIGN PATENT APPLICATION</b>  <input type="checkbox"/> Declaration Submitted with Initial Filing    OR <input checked="" type="checkbox"/> Declaration Submitted after Initial Filing	0010/PTO Rev. 6/95	U.S. Department of Commerce Patent and Trademark Office	Attorney Docket Number	H 3497 PCT/US
	First Named Inventor			Klein, Johann
	COMPLETE IF KNOWN			
	Application Number			09/856,236
	Filing Date			
	Group Art Unit			
Examiner Name				

As a below named inventor, I hereby declare that:

My residence, post office address, and citizenship are as stated below next to my name

I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled:

**PREPARATION CONTAINING GYPSUM FROM FLUE GAS DESULPHURISATION, METHOD FOR USING THE SAME AND USE THEREOF**

(Title of the invention)

the specification of which

☐ is attached hereto

OR

☒ was filed on (MM/DD/YYYY) **11/10/1999** as United States Application Number or PCT International

Application Number **PCT/EP99/08645** and was amended on (MM/DD/YYYY) \_\_\_\_\_ (if applicable).

I hereby state that I have reviewed and understand the contents of the above identified specification, including the claims, as amended by any amendment specifically referred to above.

I acknowledge the duty to disclose information which is material to patentability as defined in Title 37 Code of Federal Regulations, § 1.56.

I hereby claim foreign priority benefits under Title 35, United States Code §119(a)-(d) or §365(b) of any foreign application(s) for patent or inventor's certificate, or §365(a) of any PCT International application which designated at least one country other than the United States of America, listed below and have also identified below, by checking the box, any foreign application for patent or inventor's certificate, or of any PCT International application having a filing date before that of the application on which priority is claimed.

Prior Foreign Application Number(s)	Country	Foreign Filing Date (MM/DD/YYYY)	Priority		Certified Copy Attached?	
			Not Claimed		YES	NO
198 53 006.4	Germany	11/17/1998	<input type="checkbox"/>		<input type="checkbox"/>	<input checked="" type="checkbox"/>
			<input type="checkbox"/>		<input type="checkbox"/>	<input type="checkbox"/>
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			<input type="checkbox"/>		<input type="checkbox"/>	<input type="checkbox"/>
			<input type="checkbox"/>		<input type="checkbox"/>	<input type="checkbox"/>
			<input type="checkbox"/>		<input type="checkbox"/>	<input type="checkbox"/>

☐ Additional foreign application numbers are listed on a supplemental priority sheet attached hereto:

I hereby claim the benefit under Title 35, United States Code §119(e) of any United States provisional application(s) listed below.

Application Number(s)	Filing Date (MM/DD/YYYY)	<input type="checkbox"/> Additional provisional application numbers are listed on a supplemental priority sheet attached hereto.

Burden Hour Statement: This form is estimated to take .4 hours to complete. Time will vary depending upon the needs of the individual case. Any comments on the amount of time you are required to complete this form should be sent to the Chief Information Officer, Patent and Trademark Office, Washington, DC 20231. DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. SEND TO: Assistant Commissioner for Patents, Washington DC 20231.

**DECLARATION****Page 2**

I hereby claim the benefit under Title 35, United States Code §120 of any United States application(s), or §365© of any PCT international application designating the United States of America, listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States or PCT international application in the manner provided by the first paragraph of Title 35, United States Code §112.1 acknowledge the duty to disclose information which is material to patentability as defined in Title 37, Code of Federal Regulations §1.56 which became available between the filing date of the prior application and the national or PCT international filing date of this application.

U.S. Parent Application Number	PCT Parent Number	Parent Filing Date (MM/DD/YYYY)	Parent Patent Number (if applicable)
	PCT/EP99/08645	11/10/1999	

☐ Additional U.S. or PCT international application numbers are listed on a supplemental priority sheet attached hereto.

As a named inventor, I hereby appoint the following attorney(s) and/or agent(s) to prosecute this application and to transact all business in the Patent and Trademark Office connected therewith:

☐ Firm Name  Customer Number  or label

OR

☒ List Attorney(s) and/or agent(s) name and registration number below:

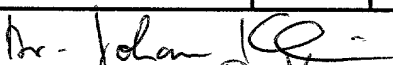
Name	Registration Number	Name	Registration Number
Wayne C. Jaeschke	21,062		
Glenn E. J. Murphy	33,539		
Stephen D. Harper	33,243		
Kimberly R. Hild	39,224		

☐ Additional attorney(s) and/or agent(s) named on a supplemental sheet attached hereto.

Please direct all correspondence to: ☒ Customer Number  or label **00423** OR ☒ Fill in correspondence address below

Name	Kimberly R. Hild				
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City	Gulph Mills	State	PA	Zip	19406
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I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

<b>Name of Sole or First Inventor:</b>		<input type="checkbox"/> A petition has been filed for this unsigned inventor			
Given Name	Johann	Middle Initial		Family Name	Klein
Inventor's Signature				Date	August 10, 2001
Residence: City	Duesseldorf	State		Country	Germany
Post Office Address	Urdenbacher Acker 20a				
Post Office Address					
City	40593 Duesseldorf	State		Zip	
		Country	Germany	Applicant Authority	

☒ Additional inventors are being named on supplemental sheet(s) attached hereto

Type a plus sign (+) inside this box → ☐

H 3497 PCT/US

DECLARATION				ADDITIONAL INVENTOR(S) Supplemental Sheet			
Name of Additional Joint Inventor, if any:				<input type="checkbox"/> A petition has been filed for this unsigned inventor			
Given Name	Gaby	Middle Initial		Family Name	Schilling	Suffix e.g. Jr.	
Inventor's Signature	<i>Gaby Schilling</i>				Date	August 10, 2001	
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Post Office Address	Bilker Allee 43						
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						Applicant Authority	
Name of Additional Joint Inventor, if any:				<input type="checkbox"/> A petition has been filed for this unsigned inventor			
Given Name	Sabine	Middle Initial		Family Name	Sipmann	Suffix e.g. Jr.	
Inventor's Signature	<i>Sabine Sipmann</i>				Date	August 10, 2001	
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Name of Additional Joint Inventor, if any:				<input type="checkbox"/> A petition has been filed for this unsigned inventor			
Given Name	Ralph	Middle Initial		Family Name	Schuetze	Suffix e.g. Jr.	
Inventor's Signature	<i>Ralph Schuetze</i>				Date	16. 7. 01	
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						Applicant Authority	
Name of Additional Joint Inventor, if any:				<input type="checkbox"/> A petition has been filed for this unsigned inventor			
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Inventor's Signature	<i>Friedhelm Koepnick</i>				Date	16. 7. 2001	
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						Applicant Authority	
<input checked="" type="checkbox"/> Additional inventors are being named on supplemental sheet(s) attached hereto							

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DECLARATION				ADDITIONAL INVENTOR(S) Supplemental Sheet			
Name of Additional Joint Inventor, if any:				<input type="checkbox"/> A petition has been filed for this unsigned inventor			
Given Name	<u>Helmut</u>	Middle Initial		Family Name	<u>Loth</u>	Suffix e.g. Jr.	
Inventor's Signature	<u>Helmut Loth</u>				Date	<u>August 10, 2007</u>	
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Given Name	<u>Klaus</u>	Middle Initial		Family Name	<u>Helpenstein</u>	Suffix e.g. Jr.	
Inventor's Signature	<u>Klaus Helpenstein</u>				Date	<u>August 10, 2007</u>	
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						Applicant Authority	
Name of Additional Joint Inventor, if any:				<input type="checkbox"/> A petition has been filed for this unsigned inventor			
Given Name	<u>Wolfgang</u>	Middle Initial		Family Name	<u>Klauck</u>	Suffix e.g. Jr.	
Inventor's Signature	<u>Wolfgang Klauck</u>				Date	<u>August 10, 2007</u>	
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Post Office Address							
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						Applicant Authority	
Name of Additional Joint Inventor, if any:				<input type="checkbox"/> A petition has been filed for this unsigned inventor			
Given Name	<u>Claudia</u>	Middle Initial		Family Name	<u>Mai</u>	Suffix e.g. Jr.	
Inventor's Signature	<u>Claudia Mai</u>				Date	<u>August 10, 2007</u>	
Residence: City	<u>Duesseldorf</u>	State		Country	<u>Germany</u>	Citizenship	<u>Germany</u>
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Post Office Address							
City	<u>40591 Duesseldorf</u>	State		Zip		Country	<u>Germany</u>
						Applicant Authority	
<input type="checkbox"/> Additional inventors are being named on supplemental sheet(s) attached hereto							